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TECHNICAL NOTE 3117

LUBRICANTS OF REDUCED FLAMMABILITY

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SUMMARY

Previous work on the correlation of structure with ease of spontane-
ous ignition for a variety of organic compounds has been summarized in
earlier reports. These studies showed that the hydrogenated polyiso-
butylenes possess outstanding resistance to spontaneous ignition, pre-
sumably as the result of the large percentage (about 80 percent) of
primary bonds of carbon to hydrogen. In this respect they are superior
to the glycol ethers, dibasic acid esters, certain silicates and phos-
phonates, and other types of hydrocarbon lubricants. They are equivalent
to the methyl siloxanes and, of the substances tested, inferior only to
the highly halogenated hydrocarbons and tricresyl phosphate; these latter
types are, of course, highly corrosive at elevated temperatures.

Determination of the change in spontaneous ignition temperature with
composition for blends of hydrogenated polyisobutylene with typical ester,
hydrocarbon, and polyether lubricants has shown that 40 percent hydrogen-
ated polyisobutylene by volume raises the ignition temperatures of these
lubricants by 60° to 80° C. Preliminary stability tests indicated that
the hydrogenated polyisobutylenes possess reasonable thermal stability,
suffering a weight loss of about 3 percent after 10 hours at 195° C
(383° F) in an inert atmosphere. Introduction of oxygen accelerates
this decomposition rate, but addition of an antioxidant (phenyl-beta-
naphthylamine) reduces the loss to 1 or 2 percent.

A practical method for synthesizing polyisobutylene largely in the
lubricant molecular weight range has been developed. This comprises the
polymerization of isobutylene with boron-trifluoride catalyst in isooctane
solution at 10° to 15° C. Both temperature control and solvent are neces-
sary to regulate the degree of polymerization.

INTRODUCTION

In initial work under this program (ref. 1), spontaneous ignition
temperatures were determined for some 50 pure organic compounds, and

earlier observations regarding the effects of structure on this property
were extended. The effects of a variety of additives and of selected
metal surfaces on spontaneous-ignition phenomena also were observed. In
the second phase of the program (ref. 2) major attention was directed to
compounds of interest in the lubricant field. The usefulness of the
crucible apparatus for determining spontaneous ignition temperature was
extended to these less volatile materials by employing spray injection
as well as dropwise addition to the ignition chamber. Examination of a
variety of lubricants in this manner showed that the hydrogenated polyiso-
butylenes possess outstanding resistance to spontaneous ignition, pre-
sumably as the result of the large percentage (about 80 percent) of primary
bonds of carbon to hydrogen.

Samples of hydrogenated polyisobutylene have been submitted to Wright
Field and to the Lewis Flight Propulsion Laboratory of the National
Advisory Committee for Aeronautics for more critical stability and per-
formance tests. Initial results from Wright Air Development Center on
the coking tendencies of hydrogenated polyisobutylene have been encourag-
ing. Uninhibited and inhibited samples deposited 2.3 and 28.7 milligrams
of coke, respectively, in the Pratt and Whitney coking test.

The present investigation has been concerned with the further study
of the spontaneous ignition of lubricants and potential lubricants. Major
emphasis has been placed on the hydrogenated polyisobutylenes - their
preparation, properties, and the properties of their blends.

This investigation was conducted at the University of Cincinnati
under the sponsorship and with the financial assistance of the National
Advisory Committee for Aeronautics.

APPARATUS AND TEST PROCEDURE

The apparatus used in this investigation has been described in
references 1 and 2. It comprised a heated metal block into which cruci-
bles of the various metals used in testing the lubricants were inserted.
Ignition temperatures were observed by adding a small measured amount of
the fuel to the chamber either as droplets or as a spray. As pointed out
in reference 2, spray injection is of most value with compounds in the
lubricant range, since it minimizes the vapor-pressure limitations of
these less volatile materials. Accordingly, the spray-injection procedure
was used throughout most of this work; however, duplicate determinations
frequently were conducted by dropwise addition, and a number of interest-
ing comparisons were obtained in this manner.

PRECISION

The precision of the crucible method of determining spontaneous ignition temperatures depends upon the temperature range of the ignition and upon the viscosity and volatility of the compound. The effects of these variables on the determination have been noted in references 1 and 2. For volatile compounds, the precision of the dropwise procedure is generally to within 1° to 2° C for those igniting below 300° C and to within 3° to 5° C for those igniting above 300° C. For the less volatile materials, the precision of the spray-injection procedure is only slightly less up to a viscosity range of 300 to 600 Saybolt Universal seconds at 37.8° C. At higher viscosities (i.e., lower vapor pressures) the present spray apparatus is not satisfactory.

RESULTS AND DISCUSSION

Spontaneous Ignition Temperatures of Lubricants and

Hydraulic Fluids

Table I summarizes the spontaneous-ignition-temperature data obtained on a number of different types of synthetic lubricants and hydraulic fluids tested during this program. The polyfluorochloro hydrocarbons, the Arochlors, and the tricresyl phosphate all have spontaneous ignition temperatures in the range of 600° C. All the halogenated compounds attacked the stainless-steel surface severely at these temperatures; the tricresyl phosphate was less corrosive but left a hard, lacquerlike residue. Little difference was observed between values obtained by the spray and dropwise additions with these compounds.

The 2-ethylhexyl silicate had a minimum ignition temperature of 246° C; the presence of 1 percent phenyl-beta-naphthylamine (antioxidant) raised this value about 9°. A characteristic white to brown silica residue was deposited. The closeness of these values to that of di(2-ethylhexyl) adipate (260° C) suggests that ignition in this range is to be expected of any compound containing 2-ethylhexyl groups as a major part of the molecule. Spontaneous ignition temperatures by the dropwise procedure were some 30° to 50° higher. Two additional diesters tested during this period, diisooctyl adipate and diethylene glycol benzoate 2-ethylhexoate, also ignited below 300° C by spray injection. The lowest values obtained by dropwise addition were 70° to 80° higher. The lacquerlike deposits left by these esters were lightest with the adipate.

Two of the Dow-Corning Siloxane lubricants (10 and 50 centistokes at 25° C) gave minimum spontaneous ignition temperatures of 374° and 430° C, respectively. These results were obtained with the commercial products as received and were essentially unchanged by "topping" the materials at 100° C under about a 10-millimeter pressure. It is of interest that these "silicone" lubricants, which comprise a $\left(-\overset{|}{\underset{|}{Si}}O-\right)_n$ chain with methyl substituents, ignite in a somewhat lower range than the hydrogenated polyisobutylenes, which comprise a $\left(-\overset{|}{\underset{|}{C}}-CH_2-\right)_n$ chain with methyl substituents.

Two dioctyl phosphonates from the Victor Chemical Co. ignited somewhat higher than would be expected of octyl esters of organic acids but still in a relatively low range (314° to 330° C); a heavy black crustlike residue remained. It is probable that in such a compound the phosphonic-acid content tends to prevent ignition but that its presence cannot have any major effect on groups like the octyl which normally ignite in the low-temperature range. A similar situation exists with the phosphates; thus, tricresyl phosphate ignites at about 600° C, while the trihexyl phosphate ignites at about 285° C.

Spontaneous-ignition-temperature data have been reported previously on another series of lubricants including a number of diesters, glycol polyethers, and hydrocarbons (ref. 2). The hydrogenated polyisobutylenes are the only materials thus far tested which have spontaneous ignition temperatures in the 400° C range, which are not corrosive at high temperatures, and which leave little appreciable residue.

Spontaneous Ignition Temperatures of Blends

of Hydrogenated Polyisobutylene

With Other Lubricants

SAE 10 motor-oil mixtures (spray-injection).- Curves showing the change in spontaneous ignition temperature with composition for mixtures of hydrogenated polyisobutylene (36 Saybolt Universal seconds at 37.8° C) and an SAE 10 motor oil at two air-flow rates are shown in figure 1. The curves show a more nearly linear relationship than those obtained for simple hydrocarbon mixtures by dropwise addition (ref. 1). A similar leveling out of the S-shape observed by dropwise addition was obtained with heptane-isooctane mixtures; some possible reasons for these differences are discussed in the section entitled "Comparison of Dropwise- and Spray-Addition Procedures: Some Observations Regarding the Nonignition Zone."

The points plotted represent true-flame ignitions. Some "smoke-pulse" ignitions were observed at temperatures slightly lower than the curves shown. However, such determinations were not sufficiently reproducible to permit a satisfactory delineation of the smoke-pulse areas.

Di(2-ethylhexyl) adipate mixtures (spray injection).- Curves showing the change in spontaneous ignition temperature with composition for mixtures of hydrogenated polyisobutylene (36 Saybolt Universal seconds at 37.8° C) and di(2-ethylhexyl) adipate at two air-flow rates are shown in figure 2. As in figure 1, the general outline of these curves is more regular than those previously obtained with other mixtures by dropwise addition. More detailed analysis of the curves shows an interesting point of inflection near the composition comprising 50 percent ester. In general, the zero air flow (higher fuel-air ratio) results in a lower spontaneous-ignition temperature as in figure 1 and as with compositions containing more than 50 percent hydrogenated polyisobutylene shown in figure 2. However, this relationship is reversed at the higher ester contents.

The reason for this peculiar reversal is not clear but probably lies in the critical dependence of the ignition temperature on the composition of the vapor phase; this in turn is dependent on the relative amounts of the two components in the liquid and upon their vapor pressures. A further illustration of this is found in figure 3, where the same type of curves have been plotted for mixtures of a slightly lower viscosity hydrogenated polyisobutylene (32 Saybolt Universal seconds at 37.8° C) and di(2-ethylhexyl) adipate by the dropwise procedure. The first and last portions of these curves are almost superimposible on those of figure 2; that is, in both cases a high portion of ester makes for easier ignition with the leaner fuel-air ratio (air-flow rate of 125 cubic centimeters per minute), while a high proportion of hydrogenated polyisobutylene makes for easier ignition with the richer fuel-air ratio (air-flow rate of 0 cubic centimeter per minute). The point where these curves cross apparently is extremely sensitive to conditions and to the nature of the hydrogenated polyisobutylene employed. In figure 2 the curves cross at 48 percent hydrogenated polyisobutylene; in figure 3 this point lies at a liquid composition of 78 percent hydrogenated polyisobutylene.

Diisooctyl-adipate mixtures (spray injection).- The curves for the spontaneous ignition temperature versus composition for mixtures of hydrogenated polyisobutylene (32 Saybolt Universal seconds at 37.8° C) and diisooctyl adipate (fig. 4) are very similar to those obtained for the di(2-ethylhexyl)-adipate blends. Here again a high proportion of ester favors ignition of the leaner fuel-air mixtures, while a high proportion of hydrogenated polyisobutylene favors ignition of the rich mixtures. The difference between the ignition temperatures at the two fuel-air ratios is more pronounced, however, amounting to about 45° C at an 80-percent-hydrogenated-polyisobutylene composition.

Polyethylene glycol (Ucon 50-HB-55) mixtures (spray injection).-
The change in spontaneous ignition temperature with composition for
mixtures of hydrogenated polyisobutylene (36 Saybolt Universal seconds
at 37.8° C) and a polyglycol lubricant (Ucon 50-HB-55) is shown in
figure 5. The general outline of these curves is similar to that of
the curves for the blends of hydrogenated polyisobutylene and motor oil
(fig. 1). While the curves cross at two points, the previously observed
tendency of hydrogenated polyisobutylene to ignite more readily at the
higher fuel-air ratio (see fig. 1).is substantiated in figure 5.

Tetra(2-ethylhexyl)-silicate mixtures (spray injection).- The change
in spontaneous ignition temperature with composition for mixtures of
hydrogenated polyisobutylene (32 Saybolt Universal seconds at 37.8° C)
with tetra(2-ethylhexyl) silicate is shown in figure 6. Here also the
general outline is similar to that of the blends of hydrogenated polyiso-
butylene and motor oil (fig. 1).

Thermal Stability of Hydrogenated Polyisobutylenes

As a preliminary indication of the thermal stability of the hydro-
genated polyisobutylenes, the weight losses of hydrogenated-polyisobutylene
samples (viscosities of 55 and 400 Saybolt Universal seconds at 37.8° C)
at 195° C (383° F) were determined under several conditions. The weight
loss of the sample with a viscosity of 400 Saybolt Universal seconds was
determined first under a nitrogen and then under an oxygen atmosphere
(fig. 7). Under nitrogen, the product lost approximately 3 percent in
weight after 10 hours. The same sample then heated 9 hours longer under
an oxygen atmosphere suffered a 6-percent additional loss in weight. This
greater loss presumably results from a more rapid chain depolymerization
initiated by oxygen attack (ref. 3).

A similar weight loss (6 percent in 12 hours) was observed with a
sample with viscosity of 55 Saybolt Universal seconds at 195° C under an
oxygen atmosphere (fig. 8). The addition of 1 percent phenyl-beta-
naphthylamine reduced this loss to less than 2 percent in 12 hours. A
hydrogenated-polyisobutylene mixture containing 60 percent by volume poly-
ethylene glycol and 1 percent phenyl-beta-naphthylamine showed a weight
loss of 4.5 percent after 12 hours at 195° C; a similar hydrogenated-
polyisobutylene-diisooctyl-adipate mixture lost less than 2 percent under
similar conditions (fig. 9). The points obtained in this work with the
hydrogenated polyisobutylene with a viscosity of 55 Saybolt Universal
seconds were somewhat erratic compared with those for the 400-Saybolt-
Universal-second sample. This may have resulted from the presence of
more volatile components in the lower viscosity material.

Comparison of Dropwise- and Spray-Addition Procedures;
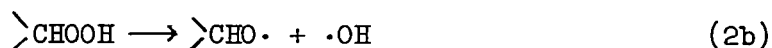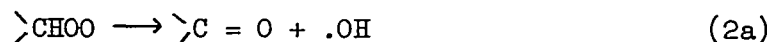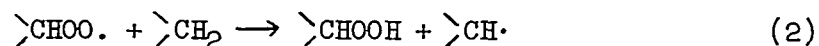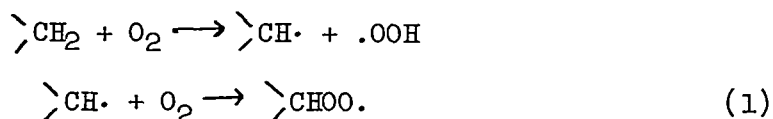
Some Observations Regarding Nonignition Zone

Characteristic S-shaped curves have been obtained in plotting the spontaneous ignition temperature (dropwise addition) against composition for a number of hydrocarbon mixtures (ref. 1). On the other hand, curves of spontaneous ignition temperature versus composition (spray injection) for various hydrogenated-polyisobutylene blends (figs. 1 to 6) have been more regular, without the large nonignition areas previously observed. Accordingly, it was of interest to prepare the corresponding curve by the spray-injection procedure for the two representative hydrocarbons, isooctane and n-heptane; the curves by the two procedures are shown in figure 10.

As in previous work, it was observed that the minimum spontaneous ignition temperature of pure volatile compounds is essentially the same by both spray and dropwise procedures. With mixtures, however, and particularly those having a large nonignition zone, differences between the results by the two procedures are quite striking. In view of the extreme sensitivity of spontaneous ignition temperatures to fuel-air ratio in the nonignition range, it is not surprising that these differences should exist. For example, it has been observed frequently that the nonignition zone for a pure compound may be completely obliterated by an increase in fuel-air ratio (refs. 1 and 4). Spray injection, being a most effective way of increasing fuel-air ratio, also is most effective in suppressing the nonignition zone as evidenced by figure 10.

This observed difference between the nonignition zones obtained by these two procedures perhaps can be explained as follows: There are two time factors involved in achieving a fuel-air mixture capable of undergoing spontaneous ignition. One is the time required to obtain the correct mixture by physical means; the other is the actual chemical induction period necessary to develop a concentration of active centers sufficient to propagate a chain-branching reaction. The first of these is not encountered in experiments which inject the premixed gases into the ignition chamber. In a spontaneous-ignition determination such as that employed here, however, the time of mixing is a very considerable factor, particularly with dropwise addition. If the chemical induction period is shorter than the time required to achieve an optimum mixture, then the active centers will tend to exhaust themselves without ever arriving at high-enough concentrations to result in a chain-branching reaction. The complex interplay between these two times with change in vapor pressure and change in structure also appears a logical explanation of the observation that the minimum spontaneous ignition temperature may sometimes be more readily achieved by dropwise than by spray addition. In such instances, the optimum conditions for spontaneous ignition apparently lie on the side of the lean fuel-air ratio, and it is easier to achieve those conditions by dropwise than by spray addition.

While the above discussion appears to be in line with observed changes in the nonignition zone, it is of interest to consider further the more fundamental reason for the existence of such a zone. This can be visualized best by first noting the most probable oxidation mechanisms involved:

$$>CH_2 + O_2 \longrightarrow >CH\cdot + \cdot OOH$$

$$>CH\cdot + O_2 \longrightarrow >CHOO\cdot \tag{1}$$

$$>CHOO\cdot + >CH_2 \longrightarrow >CHOOH + >CH\cdot \tag{2}$$

$$>CHOO \longrightarrow >C = O + \cdot OH \tag{2a}$$

$$>CHOOH \longrightarrow >CHO\cdot + \cdot OH \tag{2b}$$

Reactions (1) and (2) are the basic low-temperature reaction chain. Reactions (2a) and (2b) are subsequent reactions of the peroxy radical and of the peroxide, leading in one case to a simple chain-sustaining reaction and in the other to chain branching. It appears that the nonignition zone represents that temperature range where the peroxy radical becomes so unstable that the chain-sustaining reaction (2a) takes precedence over the chain-branching reaction (2b). As the temperature increases further the range where reaction no longer is dependent on radicals such as R· and ·OH to sustain itself is reached. (See ref. 5.) In this range the oxygen molecule is becoming sufficiently reactive to play a dominant role; this occurrence is in accord with the dependence of the high-temperature reaction on oxygen concentration and with the shortness of the induction period leading to violent reaction under those conditions.

Preparation of Hydrogenated Polyisobutylenes

The boron-trifluoride-catalyzed polymerization of isobutylene without solvent yields only about 15 percent of the product in the viscosity range of 25 to 730 Saybolt Universal seconds at $37.8°$ C (see fig. 11). In petroleum-ether solution this yield was increased to 30 percent, but the boiling-point curve suggested that ionic chain transfer between the growing polymer chains and the solvent resulted in some modification of the polymer by the solvent (fig. 12). It now has been found that the use of a solvent with proper temperature control gives over 55 percent yield of a polymer having an average viscosity of about 40 Saybolt Universal seconds at $37.8°$ C; isooctane was used as the solvent to insure that any chain transfer occurring would not affect the structure of the polyisobutylene chain. There is little doubt that further refinements in the procedure would further improve the yield of product in the lubricant range and, accordingly, that a practical synthesis could be

developed. A typical synthesis employed in preparing the hydrogenated-polyisobutylene oils employed in the present work is described below.

Isooctane (1,400 cubic centimeters) was placed in a 5-liter Erlenmeyer flask fitted with a stirrer, two gas inlet tubes, and a thermometer immersed in the liquid. The flask temperature was held at 10° to 15° C by cooling in an ice-salt bath, while 1,890 cubic centimeters of isobutylene (Phillips, 99 mole percent pure) and catalytic amounts of boron trifluoride were passed into the isooctane solution concurrently. As the reaction is strongly exothermic, the time of addition (in this case, about 8 hours) is largely dependent upon the capacity of the cooling system. The product (after being washed with dilute sodium hydroxide and water, then dried) had a volume of 3,080 cubic centimeters.

The isooctane (1,315 cubic centimeters) was recovered by distillation and the lower polymers (di-, tri-, and some tetraisobutylene) were then removed under reduced pressure. This fraction was distilled to a temperature of 125° C at 50-millimeter pressure with a distilled volume of 465 cubic centimeters. The higher polymer remaining had a volume of 1,045 cubic centimeters, a refractive index $\left(n_D^{25}\right)$ of 1.4503, and a viscosity of approximately 40 Saybolt Universal seconds at 37.8° C.

The crude product was hydrogenated in an Aminco pressure reactor using a Raney nickel catalyst and a pressure of 1,700 pounds per square inch. The resulting material gave negative tests for unsaturation. Distillation under reduced pressure yielded a series of products similar to those shown in figure 11.

SUMMARY OF RESULTS

The following results were obtained from an investigation of the ignition of lubricants of reduced flammability:

1. Spontaneous ignition temperatures have been determined for a variety of synthetic lubricants and hydraulic fluids by the spray-injection procedure. Highly halogenated hydrocarbons, the Arochlors, and tricresyl phosphate all have values in the range of 600° C. The silicates, silicones, and phosphonates tested ignited in the 250°-to-400° C range, the value depending largely upon the nature of the alkyl groups present. The hydrogenated polyisobutylenes remain the only materials which have consistently given ignition temperatures in the 400° C range, which are not corrosive at high temperatures, and which leave little appreciable residue.

2. The variation in spontaneous ignition temperature with composition has been determined for blends of hydrogenated polyisobutylene with two dibasic acid esters, a polyglycol ether, a conventional motor oil, and a silicate ester. While the detailed nature of the curves varies with the second component and with the ratio of fuel to air, the most nearly consistent effect has been an increase of 60° to 80° C over the spontaneous ignition temperature of the major component at a hydrogenated polyisobutylene content of about 40 percent.

3. The thermal stability of hydrogenated polyisobutylenes in the presence and absence of oxygen at 195° C has been determined. With 1 percent phenyl-beta-naphthylamine as a stabilizer, a typical hydrogenated polyisobutylene lost less than 2 percent by weight in 12 hours at 195° C.

4. Additional information has been obtained on the nature of the nonignition zone and on its significance as observed in the determination of spontaneous ignition temperature.

5. A practical method has been developed for the preparation of polyisobutylenes largely in the lubricant molecular-weight range. This method involves the polymerization of isobutylene using boron-trifluoride catalyst in isooctane solution at 10° to 15° C.

University of Cincinnati,
      Cincinnati, Ohio, February 4, 1953.

REFERENCES

1. Frank, Charles E., and Blackham, Angus U.: Investigation of Hydrocarbon Ignition. NACA TN 2549, 1952.

2. Frank, Charles E., Blackham, Angus U., and Swarts, Donald E.: Investigation of Spontaneous Ignition Temperatures of Organic Compounds with Particular Emphasis on Lubricants. NACA TN 2848, 1952.

3. Taylor, H.S., and Tobolsky, A. V.: Radical Chain Processes in Vinyl and Diene Reactions. Jour. Am. Chem. Soc., vol. 67, no. 12, 1945, pp. 2063-2067.

4. Sortman, C. W., Beatty, H. A., and Heron, S. D.: Spontaneous Ignition of Hydrocarbons. Ind. and Eng. Chem., vol. 33, no. 3, Mar. 1941, pp. 357-360.

5. Minkoff, G. J.: Comment on Paper "Measurement of Fast Reactions of Atomic Hydrogen" by J. C. Robb and H. W. Melville. Discussions Faraday Soc., vol. 2, 1947, pp. 147-148.

TABLE I

SPONTANEOUS IGNITION TEMPERATURES OF LUBRICANTS AND HYDRAULIC FLUIDS

Material	Spontaneous ignition temperature, °C				Remarks
	Spray injection and air flow (cm³/min) of -		Dropwise addition and air flow (cm³/min) of -		
	125	0	125	0	
(2-ethylhexyl) silicate[a] (Orsil B.F-1; Oronite Chemical Co.)	246	270	[b]300	[b]300	Fine, white silica residue; hard layer
(2-ethylhexyl) silicate + 1 percent phenyl-beta-naphthylamine (Orsil B.F-1-S; Oronite Chemical Co.)	255	253	[b]300	[b]300	Hard brown-white silica residue
Dimethyl siloxane[a] (10 centistokes at 25° C) (DC-200 series; Dow Corning Corp.)	373	372	374	374	Network of brown-white silica formed and removed periodically to obtain minimum spontaneous ignition temperature; oily residue
Dimethyl siloxane (50 centistokes at 25° C) (DC-200 series; Dow Corning Corp.)	430	433	445	447	Hard silica residue and network at high temperatures; glasslike residue at lower temperatures
Diisooctyl adipate (Plexol-244; Rohm & Haas Co., Inc.)	287	304	366	378	Slight tarnish
Diethylene glycol benzoate 2-ethylhexoate (Hooker Electrochemical Co.)	272	277	340	350	Brown-black lacquerlike residue
Dioctyl isooctene phosphonate (Victor Chemical Works)	329	330	320	319	Heavy black crustlike residue
Dioctyl benzene phosphonate (Victor Chemical Works)	316	316	314	315	Heavy black crustlike residue
Trihexyl phosphate (Shell Development Co.)	286	287	---	---	Powdery carbonaceous residue
Tricresyl phosphate[a] (Monsanto Chemical Co.)	600	600	[b]600	[b]600	Lacquerlike residue, blue metallic luster
Arochlor - 1248 (Monsanto Chemical Co.)	640	638	[b]640	[b]640	Metal badly oxidized and flaked
Arochlor - 1254 (Monsanto Chemical Co.)	595	586	595	586	Metal badly oxidized and flaked
Trichloro-1-(pentafluoroethyl) -4-(trifluoromethyl) benzene[c]	568	568	(d)	(d)	Metal badly oxidized and flaked; no noticeable residue
Dichloro-1-(chlorotetrafluoroethyl) -4-(trifluoromethyl) benzene[c]	591	587	(d)	(d)	Metal badly oxidized and flaked; no noticeable residue
3-chloro-(trifluoromethyl) benzene[c]	654	652	(d)	(d)	Metal badly oxidized and flaked; no noticeable residue
Fluorolube F-S[c] (polytrifluorochloroethylene)	652	652	(d)	(d)	Metal badly oxidized and flaked; no noticeable residue
Tetrachloro decafluoro-heptanes[c]	658	658	(d)	(d)	Metal badly oxidized and flaked; no noticeable residue

[a]"Topped" by heating in a water bath 6 hr under vacuum.

[b]Approximate value.

[c]Supplied by Wright Air Development Center.

[d]Spontaneous ignition temperature obtained by dropwise addition is similar to that obtained by spray addition.
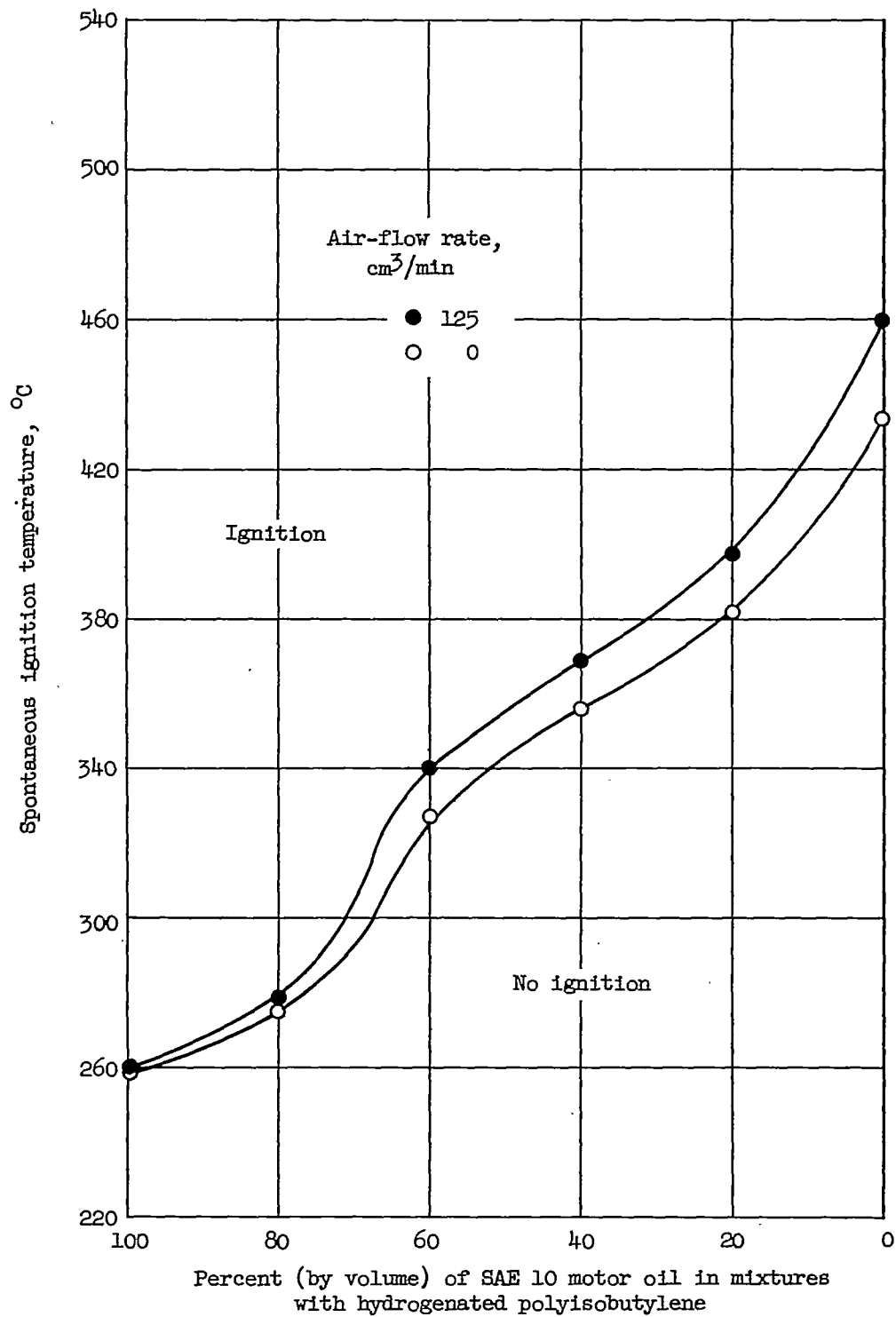
Figure 1.- Spontaneous ignition characteristics of various mixtures of SAE-10 oil and a hydrogenated polyisobutylene having a viscosity of 36 Saybolt Universal seconds. Spray-injection procedure; stainless-steel cup assembly.
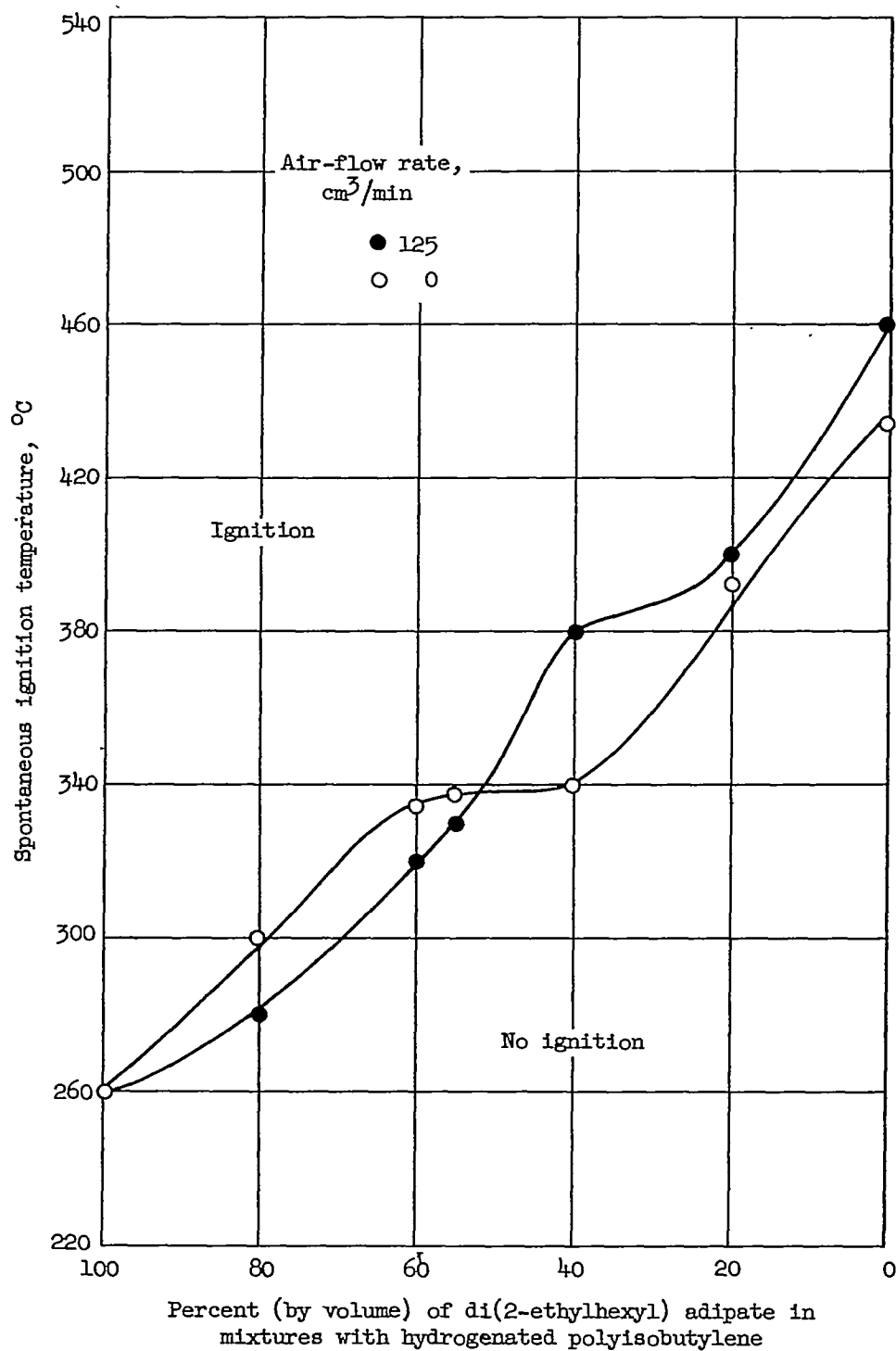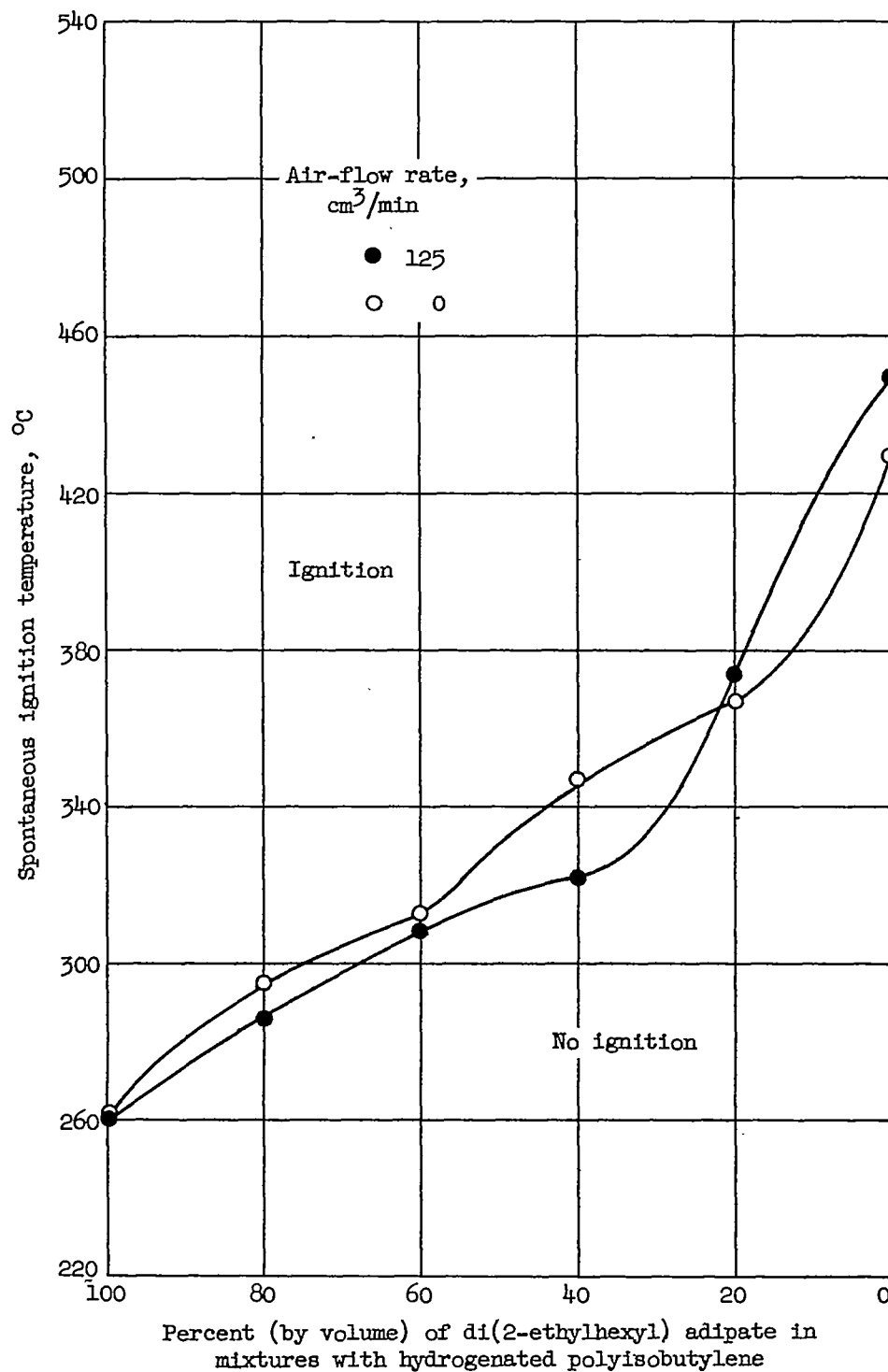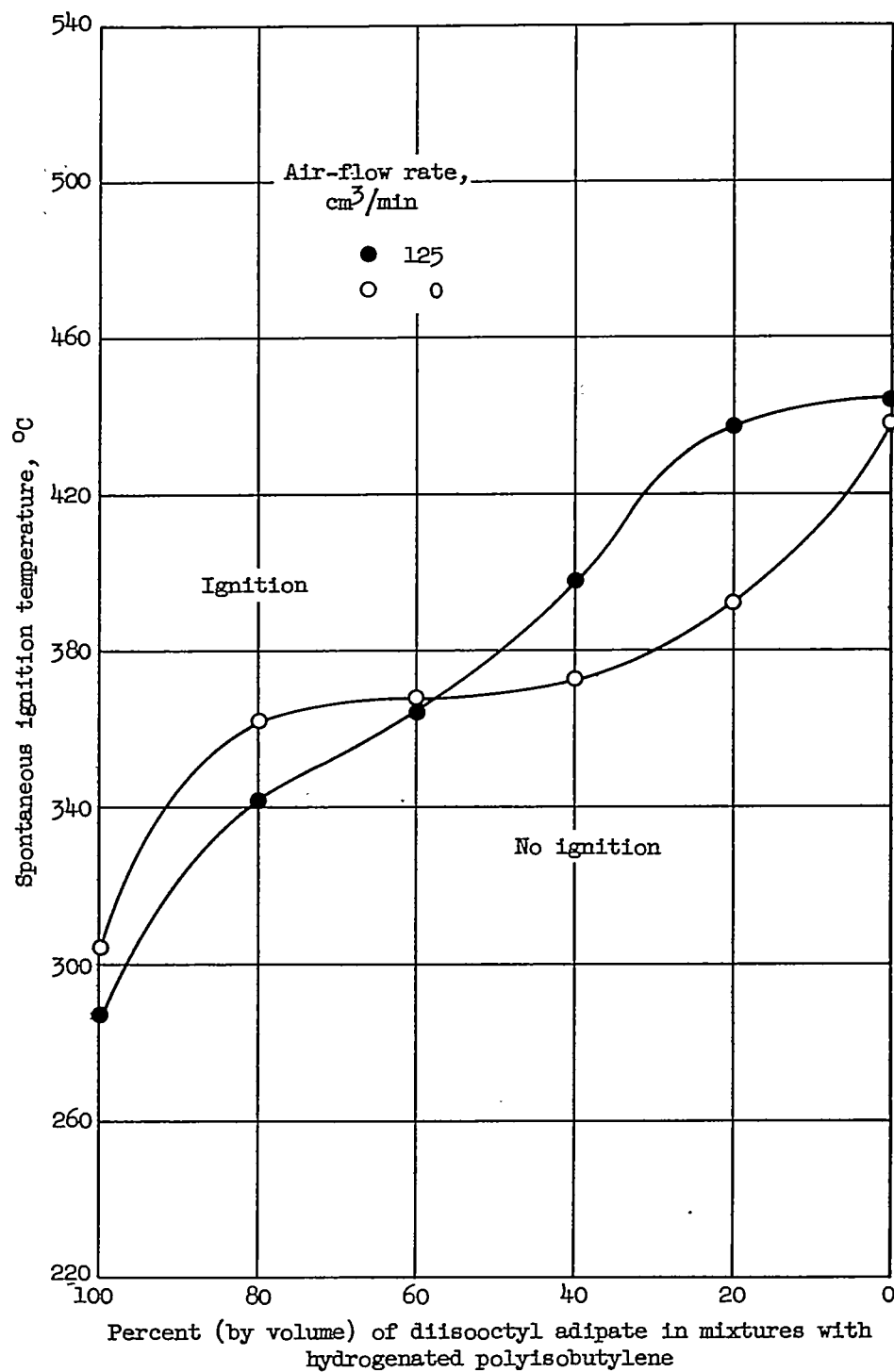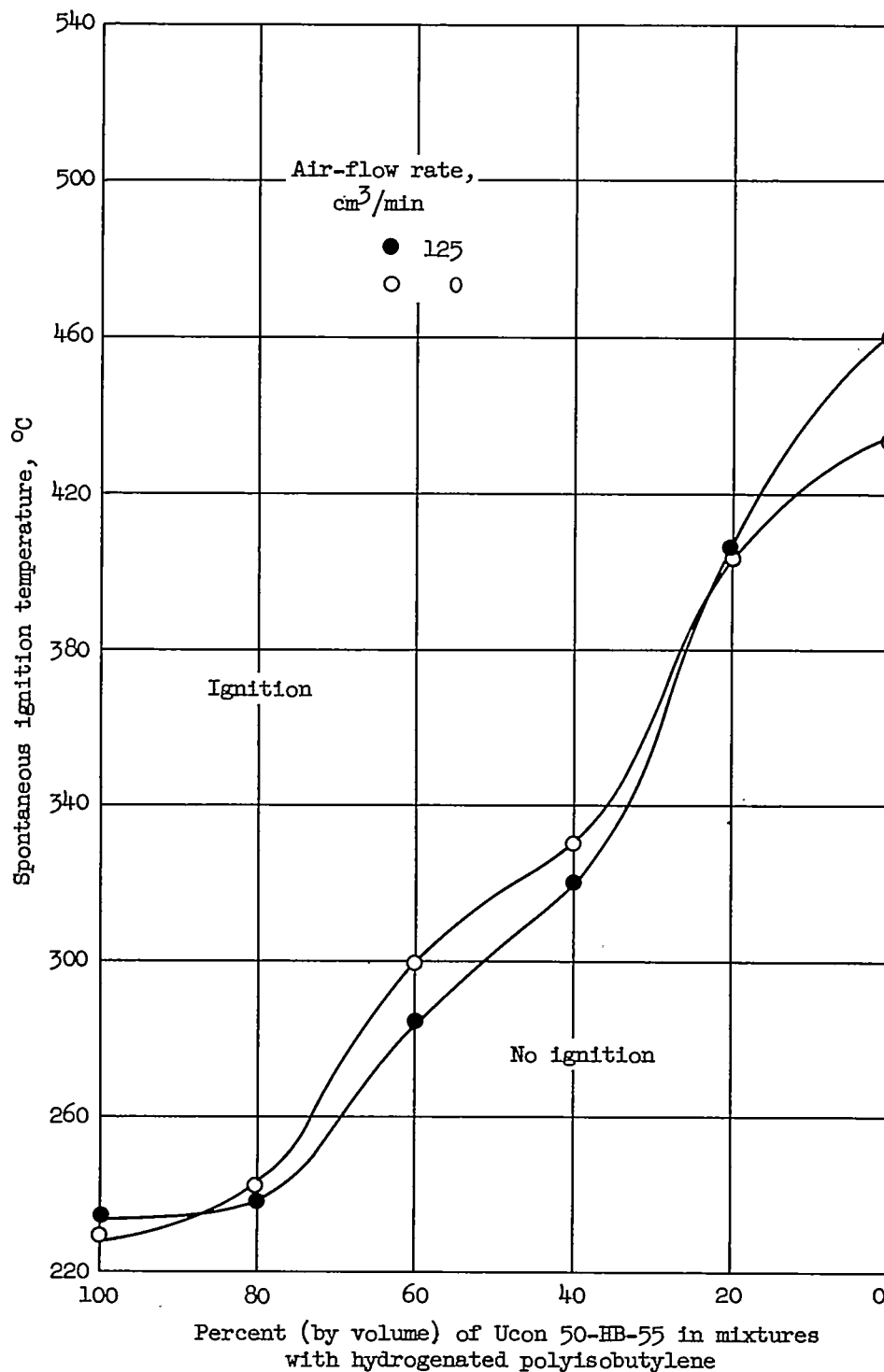
Figure 2.- Spontaneous ignition characteristics of various mixtures of
di(2-ethylhexyl) adipate and a hydrogenated polyisobutylene having a
viscosity of 36 Saybolt Universal seconds. Spray-injection procedure;
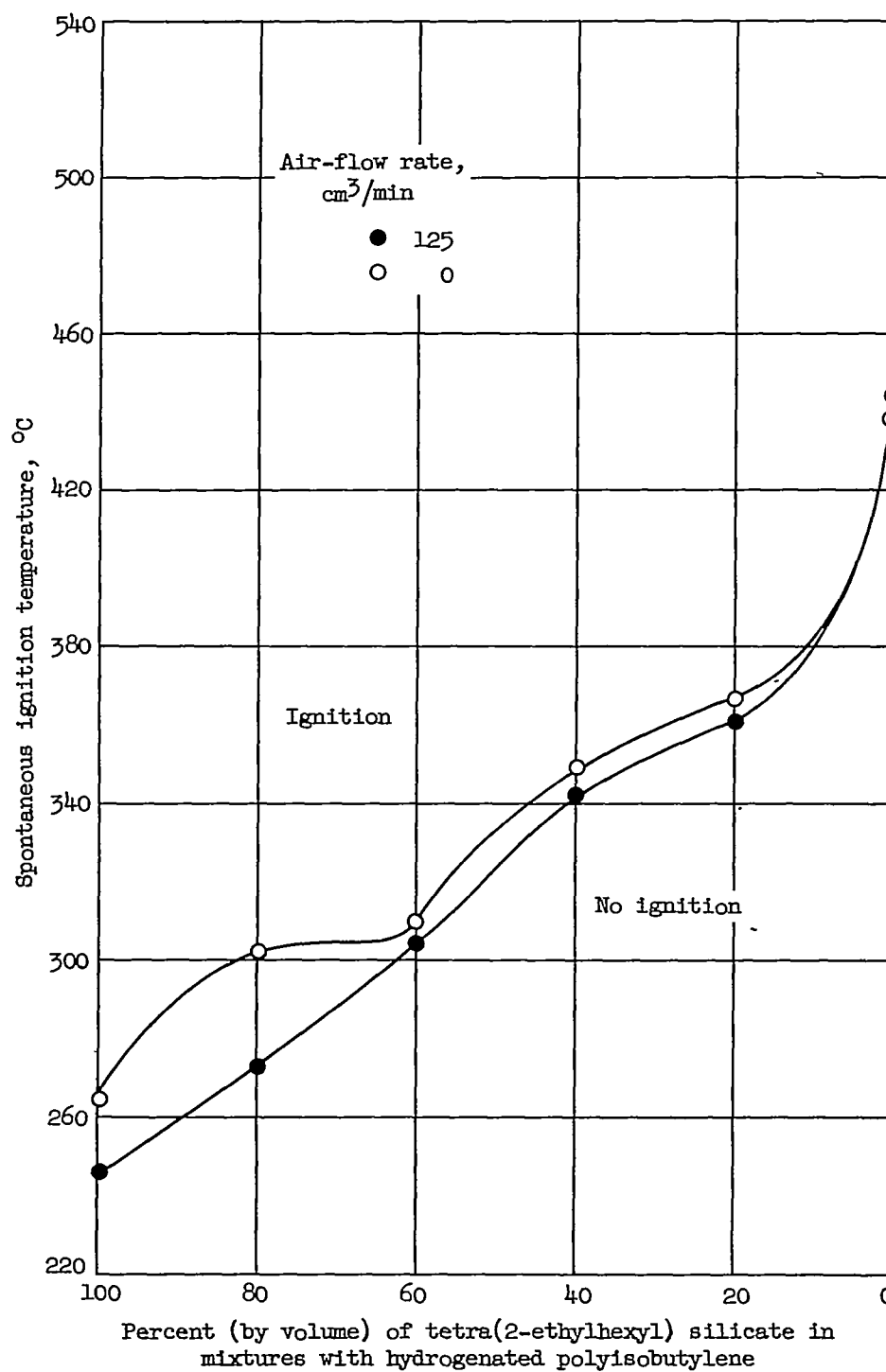stainless-steel cup assembly.

Figure 3.- Spontaneous ignition characteristics of various mixtures of di(2-ethylhexyl) adipate and a hydrogenated polyisobutylene having a viscosity of 32 Saybolt Universal seconds. Dropwise-addition procedure; stainless-steel cup assembly.

Figure 4.- Spontaneous ignition characteristics of various mixtures of
diisooctyl adipate and a hydrogenated polyisobutylene having a viscosity
of 32 Saybolt Universal seconds. Spray-injection procedure; stainless-
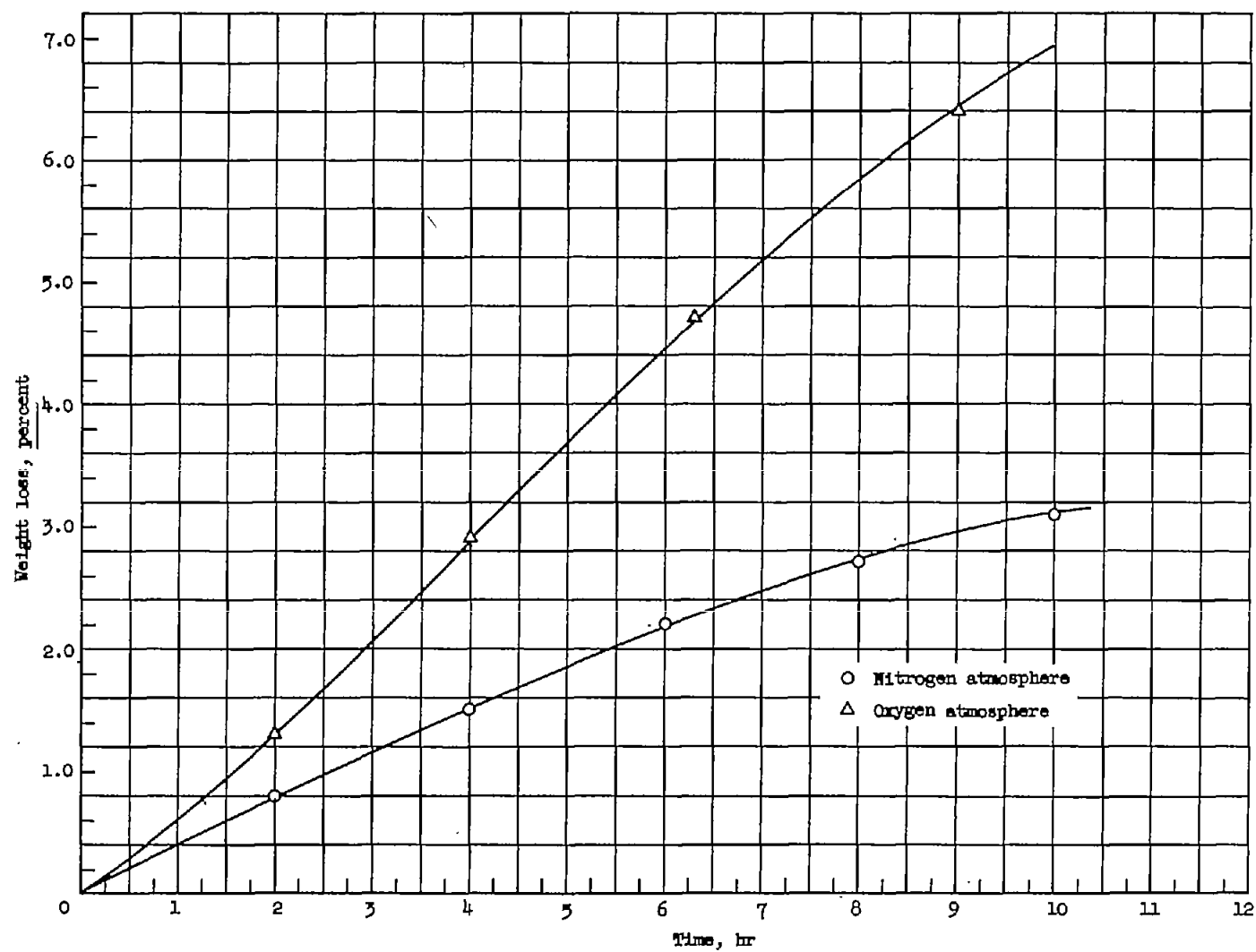steel cup assembly.

Figure 5.- Spontaneous ignition characteristics of various mixtures of
    Ucon-50-HB-55 and a hydrogenated polyisobutylene having a viscosity of
    36 Saybolt Universal seconds. Spray-injection procedure; stainless-
    steel cup assembly.

Figure 6.- Spontaneous ignition characteristics of various mixtures of
tetra(2-ethylhexyl) silicate and a hydrogenated polyisobutylene having
a viscosity of 32 Saybolt Universal seconds. Spray-injection procedure;
stainless-steel cup assembly.

Figure 7.- Thermal stability of hydrogenated polyisobutylene at 195° C.
Viscosity of sample was 400 Saybolt Universal seconds at 100° F.
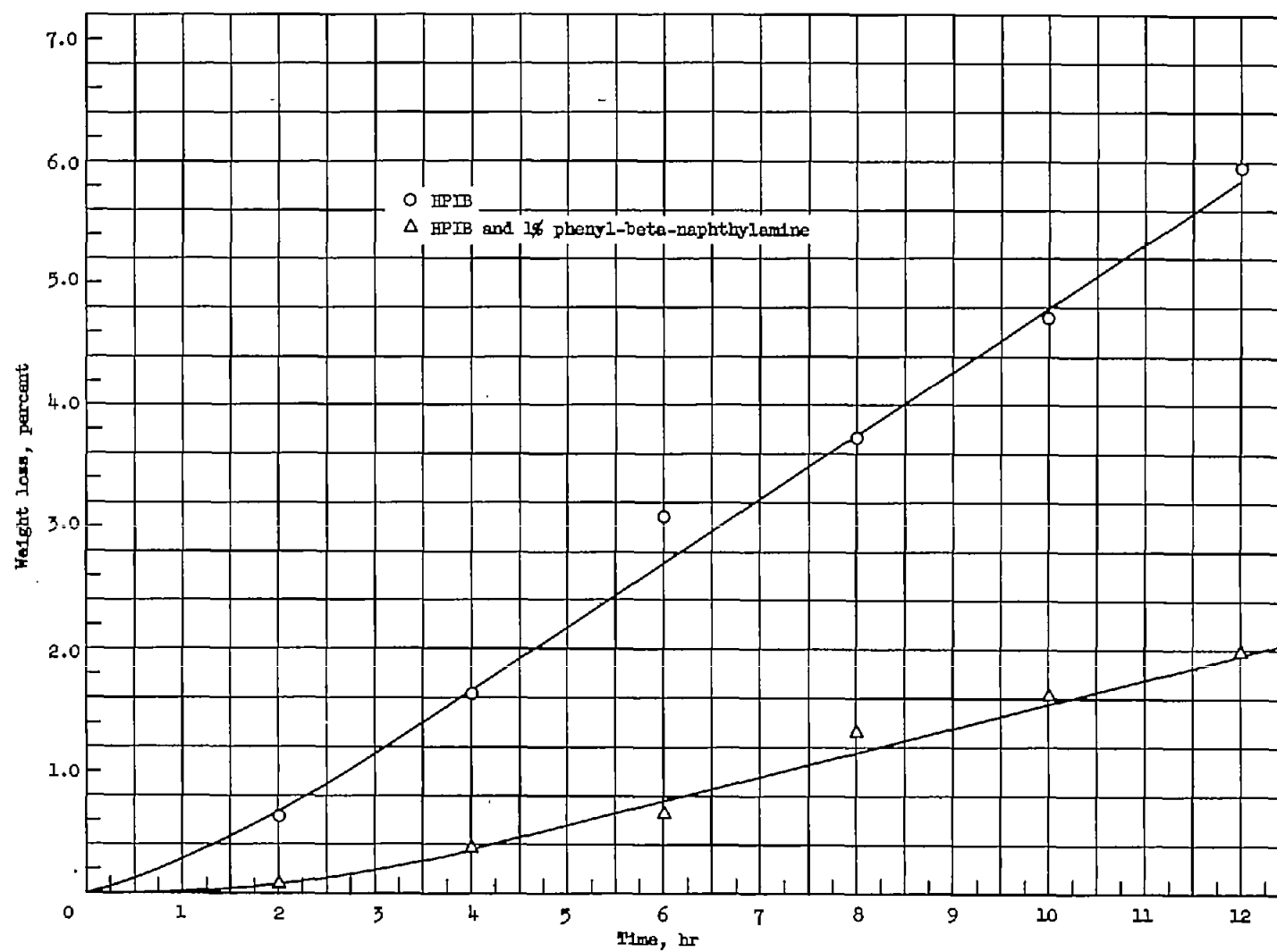
Figure 8.- Thermal stability at 195° C under oxygen atmosphere of
hydrogenated polyisobutylene (55 Saybolt Universal seconds).
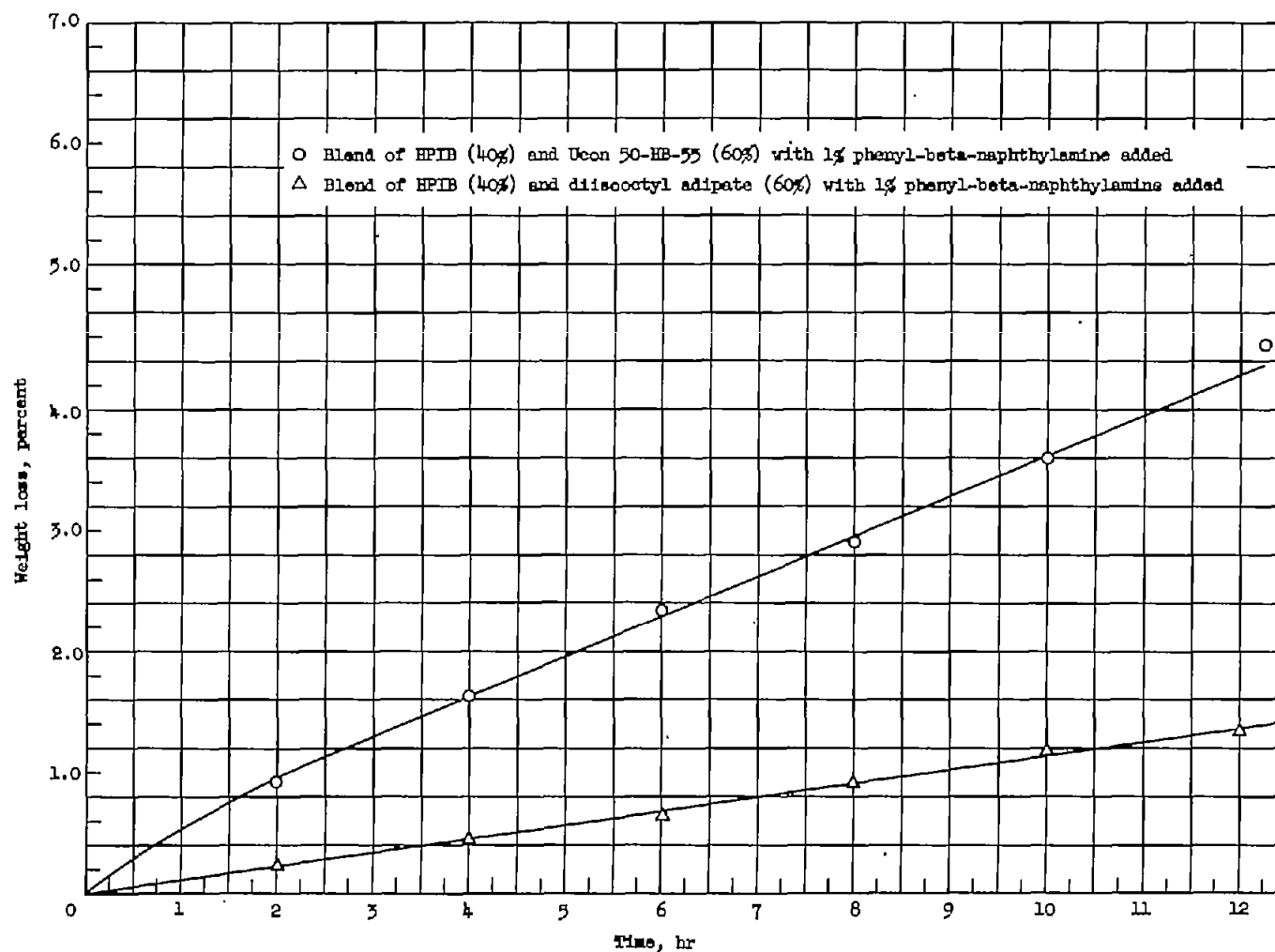
Figure 9.- Thermal stability at 195° C under oxygen atmosphere of blends
containing 40 percent hydrogenated polyisobutylene by volume (55 Saybolt
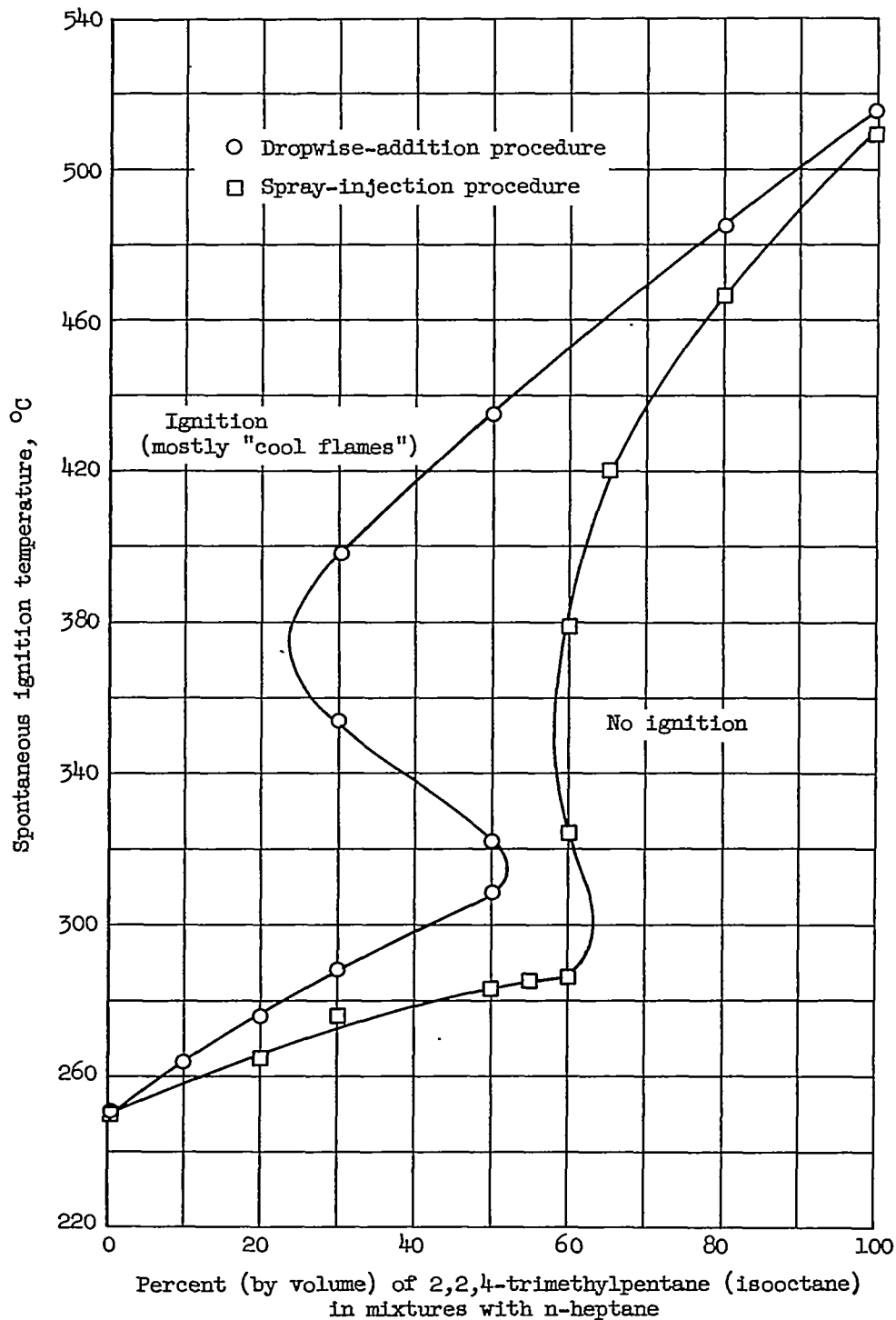Universal seconds) with 1 percent phenyl-beta-naphthylamine added.

Figure 10.- Spontaneous ignition characteristics of various mixtures of
2,2,4-trimethylpentane (isooctane) and n-heptane using two addition
procedures. Air-flow rate, 125 cubic centimeters per minute; stainless-
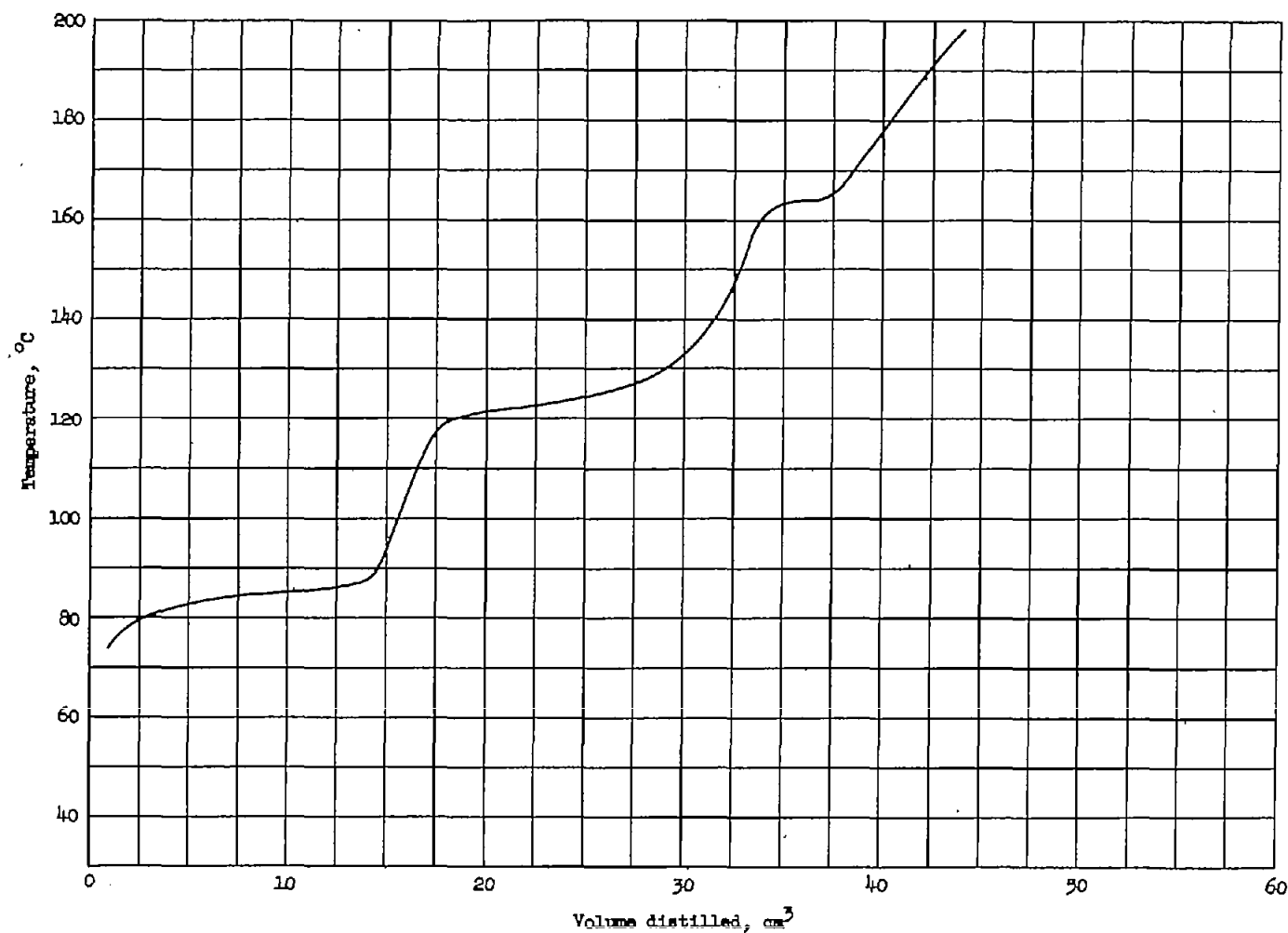steel cup assembly.

Figure 11.- Distillation at 2-millimeter pressure of hydrogenated polyisobutylene obtained from a polymerization of pure isobutylene with a boron-trifluoride catalyst in a temperature range from -40° to 60° C.
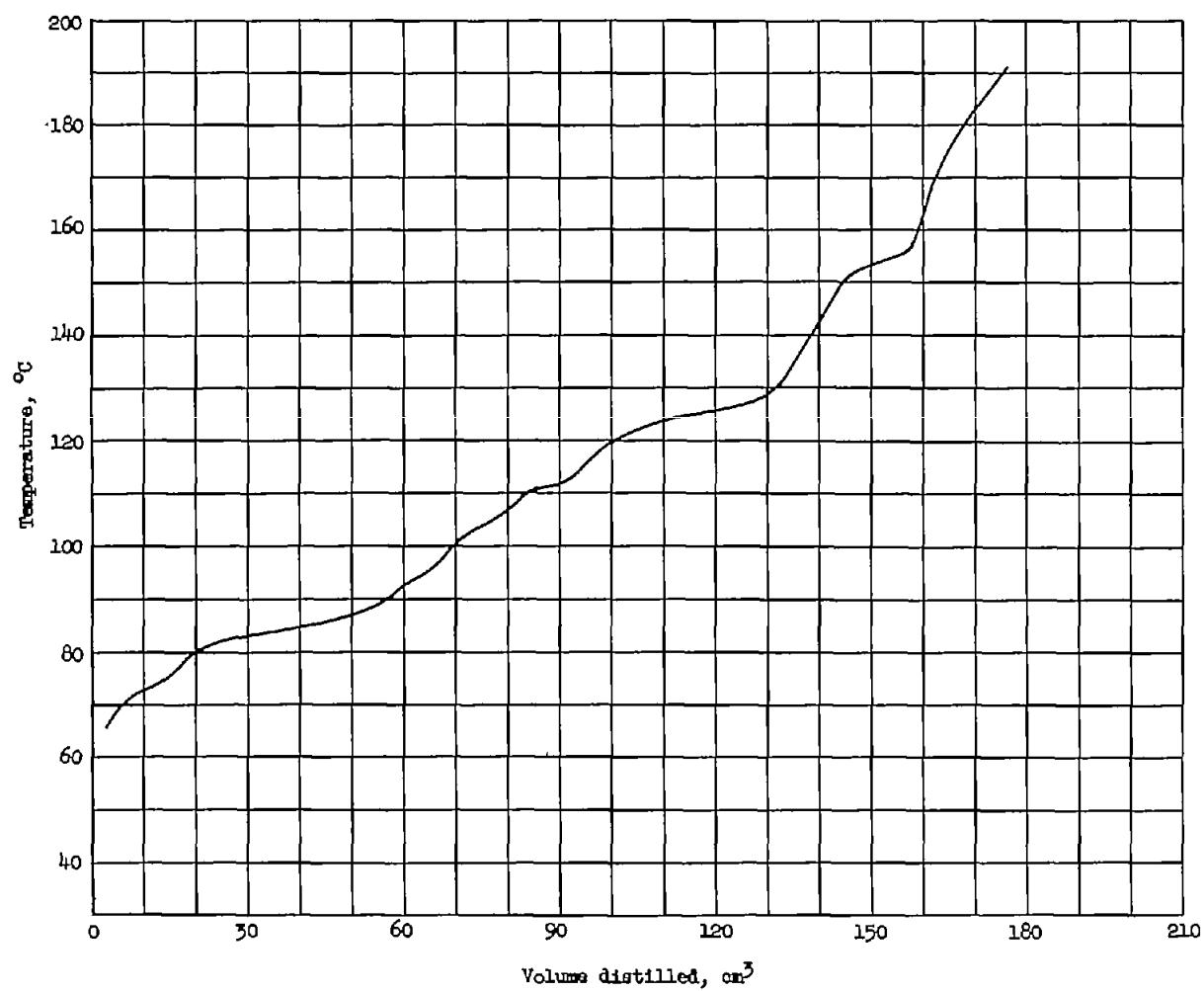
Figure 12.- Distillation at 2-millimeter pressure of hydrogenated
polyisobutylene obtained from a polymerization in 50-percent
petroleum-ether solution with temperature control. Boron-
trifluoride catalyst; temperature, -10° C.